EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

# Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF**.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
*https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do*

## Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

### Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report")*,
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

## Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

### Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

### Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

### Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

<table>
<tr><td></td><td>Experiment title:<br>Structural origin of dynamical arrest in gels and glasses</td><td>Experiment number:<br>SC-5233</td></tr>
<tr><td>Beamline:<br>ID02</td><td>Date of experiment:<br>from: 20.04.2022 to: 22.04.2022</td><td rowspan="2">Date of report:<br>18.07.2022<br>Received at ESRF:</td></tr>
<tr><td>Shifts:<br>6</td><td>Local contact(s):<br>William Chevremont ( email: william.chevremont@esrf.fr )</td></tr>
</table>

**Names and affiliations of applicants** (* indicates experimentalists):

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## Report:

When colloidal dispersions reach a particular concentration, usually quantified by the volume fraction,
they enter in an arrested state. Depending on the inter-particle interactions these states are typically associated to a gel (attractive) or glass (repulsive) phase [1]. Even tough being very similar, and in some cases composed by the same building blocks, these two classes of materials can manifest very different macroscopic properties, e.g. the mechanical response to an external stress. The amorphous soft materials can be found in many practical applications, being relevant in several industries ranging from medical and food to optoelectronic, but the lack of a fundamental coherent picture of these phenomena means that they are often treated with ad-hoc explanations. The consequence is that the reasons that produce these dynamically arrested amorphous systems are known only qualitatively [2], and a complete understanding of the general mechanisms are still lacking. Grasping the structural origins of the dynamical arrest in disordered systems is one of the long-standing goals of condensed matter physics, which is finally on sight with new theoretical and experimental techniques. Simulations and experimental works suggest the presence of a particular intermediate range
structure, visible in higher order correlation functions, that appear to be correlated with the glass formation
process [3].

The experiment was performed at ID02 with 12keV in USAXS configuration. we measured samples that are able to change reversibly the interparticle potential, thus capable to switch between an arrested glass and an arrested gel structure. More in detail the samples were nanoparticles composed by a silica core and a PNIPAm shell. In total we measured 6 different samples at 4 different volume fractions ( plus diluted samples for the form factor). We carried out our measurements at 6 different temperatures ranging from 20 °C to 45 °C to observe the transition between the glasssy state (swollen PNIPAm shell) to the gel state (collapsed PNIPAm shell). We collected an XPCS measurement for each temperature to control the dynamical condition of the sample and we collected speckle patterns from all the capillary volume to obtain information of the intermediate range order via XCCA analysis. In figure 1 some preliminary results concerning the sole structure factor are reported for 3 samples at the same nominal concentration. The shown samples have the same thickenss of the PNIPAm shell and differ only in the amount of cross-linker, with the sample in figure 1.a the one characterized by the least amount of cross-linker and the sample in fig. 1.c the one with the highest.

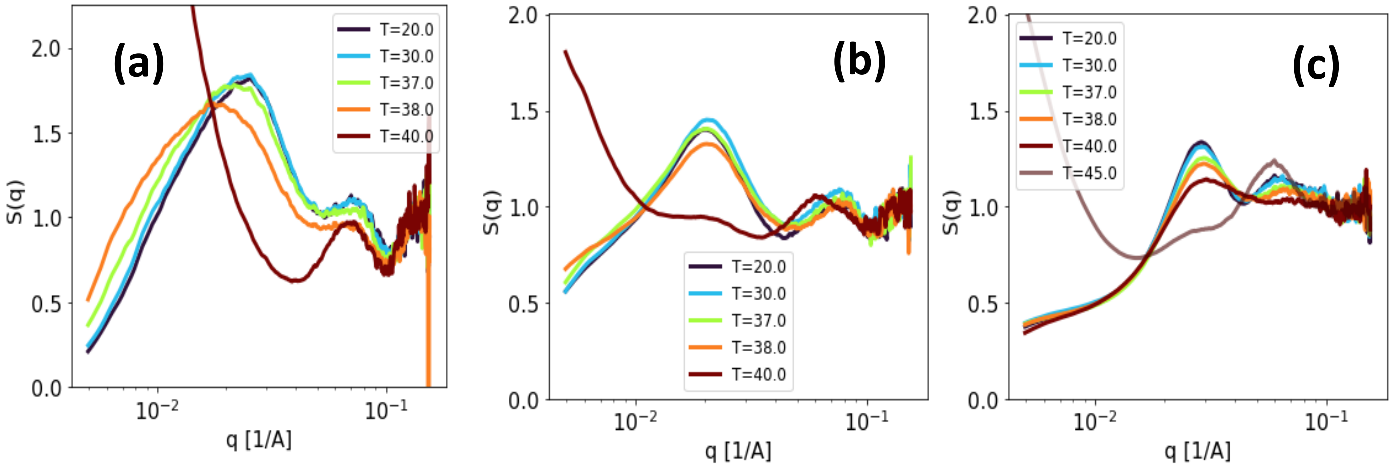


*Figure 1: transition from a glassy structure to a gel observed in 3 samples at increasing cross-linker density (from a to c).*

Currently the analysis to extract the angular cross-correlations from all the collected speckle patterns for all the investigated samples is still ongoing.

## References:


[1] K. A. Dawson, The glass paradigm for colloidal glasses, gels, and other arrested states driven by attractive interactions,
Current Opinion in Colloid & Interface Science, 7, 3, 218-227, (2002), (doi: 10.1016/S1359-0294(02)00052-3)
[2] I. Tah, A. Mutneja, and S. Karmakar, Understanding Slow and Heterogeneous Dynamics in Model Supercooled Glass-
Forming Liquids, ACS Omega, 6, 11, 7229-7239, (2021) (doi: 10.1021/acsomega.0c04831)
[3] Z. Zhang and W. Kob, Revealing the three-dimensional structure of liquids using four-point correlation functions, PNAS 117,
14032-14037 (2020) (doi:10.1073/pnas.2005638117)